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(54) Title: A METHOD FOR THE FRACTIONATION OF MOLASSES

(57) Abstract

The invention relates to a method for the fractionation of molasses using a chromatographic simulated moving bed system in which the liquid flow is effected in a single direction in a system comprising at least two chromatographic partial packing material beds. In the method of the invention, the product or products are recovered during a multi-step sequence comprising the following phases: feeding phase of molasses, eluting phase and recycling phase. The liquid present in the partial packing material beds with their dry solids profile is recycled in the recycling phase in a loop comprising one, two or several partial packing material beds.

1

A method for the fractionation of molasses

The present invention relates to a method for the
fractionation of molasses using a chromatographic simu-
lated moving bed system in which the liquid flow is
effected in a single direction in a system comprising
at least two chromatographic partial packing material
beds. Fractionation of molasses denotes fractionation
of various vegetable-derived by-products of the food and
fermenting industries, such as beet and cane molasses,
stillage, vinasse, slop, wood molasses, corn steep
water, wheat, barley and corn molasses (hydrolyzed C
starch). In the method of the invention, the product or
products are recovered during a multi-step sequence
comprising the following phases: feeding phase of molas-
ses, eluting phase and recycling phase.
        The liquid present in the partial packing mater-
ial beds with their dry solids profile is recycled in
the recycling phase in a loop comprising one, two or
several partial packing material beds.
        These phases are employed to form sequences com-
prising several process steps. In accordance with the
invention, a sequence preferably comprises five to ten
steps. A step comprises for example
        a molasses feeding phase and/or feeding of an
eluant liquid and recovery of the product or products,
or
        an eluting phase with recovery of a product or
products, or
        recycling phase and eluting phase with recovery
of a product or products, or
        two or more recycling phases.
        Sequences comprising said steps are repeated five
to seven times to reach an equilibrium.
        Typically from three to twelve, preferably three

2

to six chromatographic partial packing material beds
are employed. A loop may comprise one, two or several
partial packing material beds. Strongly acid cation
exchange resin is preferably used as the column packing
5   material.

The simulated moving bed system has been developed
and introduced by UOP (United Oil Products), U.S.A., at
the beginning of the 1960's, initially for petrochemical
applications (U.S. Patent 2,985,589). Today several
10   simulated moving bed methods for a number of different
applications are known (U.S. Patents 3,706,812,
4,157,267, 4,267,054, 4,293,346, 4,312,678, 4,313,015,
4,332,623, 4,359,430, 4,379,751, 4,402,832, 4,412,866,
4,461,649, 4,533,398 and 5,127,957, and published Euro-
15   pean application 0,279,946).

The simulated moving bed system enables separating
performances that are many times higher, and lower dilu-
tion of the products (consumption of eluant) than in the
batch method.
20   The simulated moving bed method is either con-
tinuous or sequential.

In a continuous simulated moving bed method, all
flows are continuous. These flows are: feeding of feed
solution and eluant liquid, recycling of liquid mixture
25   and recovery of products (usually only two). The flow
rate for these flows may be adjusted in accordance with
the separation goals (yield, purity, capacity). Nor-
mally, 8 to 20 partial packing material beds are com-
bined into a single loop. In accordance with the above-
30   mentioned U.S. Patent 4,402,832, the recycling phases
have been applied to the recycling of dilute fractions.
The feed and product recovery points are shifted cycli-
cally in the downstream direction. On account of the
feed of eluant liquid and feed solution (and on account
35   of recovery of products) and the flow through the packing

3

material bed, a dry solids profile is formed. Ingredi-
ents having a lower migration rate in the packing bed
are concentrated at the downstream end of the dry sol-
ids profile, and respectively ingredients having a high-
5   er migration rate at the upstream end. Feeding points
for feeding solution and eluant liquid and recovery
points for product or products are shifted gradually at
substantially the same rate at which the dry solids
profile moves in the bed. The product or products are
10   recovered substantially from the upstream and downstream
end of the dry solids profile, and the feed solution is
fed approximately to the maximum point of the dry solids
profile and the eluant liquid approximately to the mini-
mum point of the dry solids profile. Part of the separ-
15   ated product fraction is recycled on account of the
continuous cyclic flow and as only part of the dry
solids profile is removed from the packing material bed.

The cyclical shifting of the feed and recovery
points is performed by using feed and recovery valves
20   disposed along the packing material bed at the upstream
and downstream end of each partial packing material bed.
If it is desired to recover product fractions of high
purity, short phase times and a plurality of partial
packing material beds must be employed (the apparatus
25   has corresponding valves and feed and recovery equip-
ment).

In a sequential simulated moving bed method, not
all flows are continuous. In a sequential simulated
moving bed method the flows are: feeding of feed solu-
30   tion and eluant liquid, recycling of liquid mixture and
recovery of products (two to four or more products; e.g.
betaine as a third fraction in beet molasses separation
and monosaccharides in cane sugar separation). The flow
rate and the volumes of the different feeds and product
35   fractions may be adjusted in accordance with the separ-

4

ation goals (yield, purity, capacity). The method com-
prises three basic phases: feeding, elution, and recyc-
ling. During the feed phase, a feed solution and pos-
sibly also an eluant liquid is fed into predetermined
partial packing material beds, and simultaneously two
or even three product fractions are recovered. During
the eluting phase, eluant liquid is fed into a predet-
ermined partial packing material bed, and during said
phases one or even two product fractions are recovered
in addition to the residue. During the recycling phase,
no feed solution or eluant liquid is fed into the partial
packing material beds and no products are recovered.

Finnish Patent Application 882740 (U.S. Patent
5,127,957) discloses a method for recovery of betaine
and sucrose from beet molasses using a sequential simul-
ated moving bed method, the chromatographic system
therein comprising at least three chromatographic par-
tial packing material beds connected in series and
adapted for the flow of liquids in a single direction
in partial packing material beds, in which method beta-
ine and sucrose are separated during the same sequence
comprising:

        - molasses feeding phase, in which a molasses feed
solution is fed into one of said partial packing mater-
ial beds and in which eluant water is fed substantially
simultaneously into another partial packing material
bed,

        - feeding phase of eluant water, and
        - recycling phase,
        these phases being repeated either once or several
times during the sequence.

        A novel sequential simulated moving bed method has
now been developed, which is particularly suitable for
the fractionation of molasses. The novel method yields
a purer sucrose solution with a better yield and/or capa-

5

city.

In the novel method, the liquid flow is arranged in a single direction in a system comprising at least two partial packing material beds, and the product is/ products are recovered during a multi-step sequence. The partial packing material bed usually comprises one column. The sequence comprises feeding, eluting and recycling phases. During the recycling phase, the liquid in the partial packing material beds with their dry solids profile is recycled in a loop comprising one, two or several partial packing material beds.

Therefore, in the novel method recycling is employed in a novel way. In the recycling phase one, two or three or even more separate successive loops are formed. For example, the number of columns being four, the loop preferably comprises two columns. The loop may be closed or "open", i.e., when liquid is recycled in the other loop, eluant liquid can be fed into the other loop and the product fraction can be recovered therefrom. During feed and elution, the flow through the packing material beds may be effected between successive loops, the flows conveying material from one loop to another. During the recycling phase, the loop is closed and separated from the other loops. One separate dry solids profile is recycled in each loop.

Molasses is rich in various coloured components (colourants) which were difficult to remove completely enough by the earlier methods. Separate colour-removing phases were needed, or two-step crystallization had to be used to obtain a colourless product. The novel method according to the invention affords even 90% or greater colour removal in the molasses fractionating phase alone. The major part of the colour is separated already in the column group of that loop to which the feed solution is supplied, and it will not essentially contaminate the

6

column groups of the second (or third) loop. Continuous and stable colour removal is achieved. When a batch method or conventional simulated moving bed methods (continuous or sequential), for instance, are employed, colour removal is normally only 75-80% in continuous long-term separation.

Also the separation of non-sugars, i.e. salts, is efficient when the method of the invention is used, and thus the sucrose content of the sugar fraction obtained from the separation can be very high, usually in advantageous cases in excess of 92-95% on the dry solids. The majority of the salts is separated already in the column group of that loop to which the feed solution is supplied, and thus the ion exclusion is more complete in the following loops. The result is a more symmetrical, sharper and higher sucrose peak, in other words, better separation of sucrose is achieved. When the bath method or conventional simulated moving bed methods (continuous or sequential), for instance, are employed, the sucrose content of the sugar fraction is in advantageous cases usually about or below 90-92% on the dry solids.

A strongly acid, gel-type cation exchange resin (e.g. "Zerolit 225", "Finex" or "Purolite") preferably in the sodium or potassium form is used as a packing for the columns.

Prior to the chromatographic fractionation, the feed solution (beet molasses) is preferably diluted with water to 20-65% by weight, softened with sodium carbonate and finally filtered using diatomaceous earth as a filtering aid. Prior to feed into separation columns, the molasses solution is heated to 40-85°C and even to 95°C.

Water preferably at 40-85°C is used for the elution.

The flow rate of the liquid in the columns is 0.5-

7

10 m^3/h/m^2, even 20 m^3/h/m^2.

The following examples illustrate the novel sequential simulated moving bed method for the fractionation of molasses. These examples shall not be regarded as restricting the scope of the invention, as they are only examples of employing the method according to the invention to recover sucrose and betaine from beet molasses.

Example 1

A pilot plant scale chromatographic test apparatus was employed. The apparatus included four columns, feed pumps, recycling pumps, eluant water pumps, flow and pressure regulators, and inlet and outlet valves for the different process streams. The flowchart is shown in Figure 1.

The columns were packed with a strongly acid cation exchange resin ("Purolite"). The resin had a polystyrene/divinylbenzene backbone and was activated with sulphonic acid groups; the mean spherule size was about 0.36 mm. The resin had a DVB content of 5.5%. Initially the resin had been regenerated to sodium form, and during the run it was balanced with cations from the feed molasses.

Test conditions:

Diameter of columns	200 mm
Height of resin bed/ column	2800 mm
Temperature	75°C
Flow rate	40, 50, 70 and 90 l/h

The feed solution consisted of beet molasses wherefrom calcium had been removed by adding sodium carbonate (pH 9) and filtering the precipitate off using diatomaceous earth as an aid.

The separation of sucrose and betaine was performed by an eight-step sequence in which each column had its

8

specific function. As shown in Figure 1, steps 5, 6 and
7 each comprise one recycling phase and one feeding
phase for eluant water, and step 8 two recycling phases.
The duration of the sequence was 79 minutes and the

5   sucrose yield 84.0% (on the amount of sucrose fed).
Step 1: Molasses was fed (feeding phase) into col-
umn 1 at flow rate 50 l/h, and the residue fraction was
eluted from the downstream end of the column. Simultane-
ously water was supplied (eluting phase) to column 2 at

10   a flow rate 90 l/h, and a recycling fraction and sucrose
were eluted from column 4. Said recycling fraction was
used to dilute the raw material (molasses).
Step 2: Feeding of molasses into column 1 and
elution of residue from the downstream end of column 1

15   were continued. Simultaneously water was supplied to
columns 2 and 4 at a flow rate 90 l/h, the residue frac-
tion was eluted from column 3, and the elution of sucrose
was continued from column 4.
Step 3: Water was fed into columns 1 (50 l/h) and

20   4 (90 l/h), and the residue fraction was eluted from
columns 1 and 3.
Step 4: Water was fed into column 2 at a rate 90
l/h, and the residue fraction was eluted from column
3.

25   Step 5: Recycling (recycling phase) in columns 1
and 2 at a rate 90 l/h; simultaneously water was sup-
plied to column 3 at a rate 70 l/h and the betaine frac-
tion was eluted from column 4.
Step 6: Water was fed into column 1 at a rate 90

30   l/h and the residue fraction was eluted from column 2;
simultaneous recycling in columns 3 and 4 at a rate 70
l/h.
Step 7: Recycling in columns 1 and 2 at a rate 90
l/h.

35   Step 8: Recycling in columns 1 and 2 at a rate 90

l/h and in columns 3 and 4 at a rate 40 l/h.

        After the sequence was completed, the process
control program was continued and it returned to step
1. By repeating this sequence five to seven times, an
equilibrium was reached in the system. The run was con-
tinued in a state of equilibrium, and product fractions
with a constant composition were recovered and analyzed
(cf. Tables 1 and 2).

        The progress of the separation process was moni-
tored with a density meter, a meter for optical activ-
ity, and a conductivity meter, and the separation was
controlled by a microprocessor whereby precisely defined
volumes and flow rates of feeds, recycled liquid and
product fractions were controlled employing quan-
tity/volume measuring, valves and pumps.

        Table 1 shows the volumes of the feeds, recycled
liquid and product fractions, and Table 2 shows the
compositions of molasses and the product fractions. The
sucrose and betaine fractions were recovered from column
4. Table 5 shows the colours of the molasses, residues
and product fractions.

Table 1

Volumes of feeds, recycled liquid and product fractions (1)

Step No.	1	2	3	4	5	6	7	8
Molasses feed	18[x)	-	-	-	-	-	-	-
Water feed	21	5.0+8.5	4.0+8.0	5.0	26.0	25.0	0	-
Raffinate fraction from column 1	18[x)	-	4.0	-	-	-	-	-
Raffinate fraction from column 2, 3 or 4	-	5.0	8.0	5.0	-	25.0	0	-
Betaine fraction	-	-	-	-	26.0[xx)	-	-	-
Recycle fraction	7.5	-	-	-	-	-	-	-
Sucrose fraction	13.5	8.5	-	-	-	-	-	-
Recycled solution	-	-	-	-	26.0	20.0	26.0[xxx)	-

x)   Total from steps 1 and 2

xx)   Total from steps 4 and 5

xxx)   Total from steps 7 and 8

11

Table 2

Compositions of feed and product fractions

	Dry solids (kg/l)	Sucrose (% by weight on d.s.)	Betaine (% by weight on d.s.)	Other substances (% by weight on d.s.)
Molasses feed	0.76	58.2	5.6	36.2
Residue fraction	0.075	21.2	7.5	71.3
Betaine fraction	0.028	10.1	41.4	48.5
Sucrose fraction	0.279	94.8	0.7	4.5

Example 2

A pilot plant scale chromatographic test apparatus was employed. The apparatus included three columns, feed pumps, recycling pumps, eluant water pumps, flow and pressure regulators, and inlet and outlet valves for the different process streams. The flowchart is shown in Figure 2.

The columns had been packed with a strongly acid cation exchange resin ("Purolite"). The resin had a polystyrene/divinylbenzene backbone and was activated with sulphonic acid groups; the mean spherule size was about 0.36 mm. The resin had a DVB content of 5.5%. Initially the resin had been regenerated to sodium form, and during the run it was balanced with cations from the feed molasses.

Test conditions:
Diameter of columns   200 mm
Height of resin bed:
columns 1 and 3   4100 mm
column 2   2800 mm
Temperature   75°C
Flow rates   25, 35, 45, 85 and 110 l/h
The feed solution consisted of beet molasses

SUBSTITUTE SHEET

12

wherefrom calcium had been removed by adding sodium
carbonate (pH 9) and filtering the precipitate off using
diatomaceous earth as an aid.

The separation of sucrose and betaine was performed
by a five-step sequence in which each column had its
specific function. As shown in Figure 2, steps 2 and 3
each comprise one recycling phase and one feeding phase
for eluant water, and step 5 two recycling phases. The
duration of the sequence was 100 minutes and the sucrose
yield 87.3% (on the amount of sucrose fed).

Step 1: Molasses was fed into column 1 at flow rate
45 l/h, and residue was eluted from the same column
(downstream end of the column); simultaneously water was
supplied to column 2, and a recycling fraction and suc-
rose fraction were eluted from column 3 at a flow rate
85 l/h.

Step 2: Water was fed into column 2 at a rate 110
l/h, and the residue fraction was eluted from column
1; simultaneous recycling in column 3 at a rate 25 l/h.

Step 3: Recycling in columns 1 and 2 at a rate 110
l/h; simultaneously water was supplied to column 3 at
a rate 35 l/h and the betaine fraction was eluted from
the same column.

Step 4: Water was fed into column 1 at a rate 110
l/h and into column 3 at a rate 35 l/h, and the residue
fraction was eluted from columns 2 and 3.

Step 5: Recycling in columns 1 and 2 at a rate 110
l/h and in column 3 at a rate 25 l/h.

After the sequence was completed, the process
control program was continued and it returned to step
1. By repeating this sequence five to seven times, an
equilibrium was reached in the system. The run was con-
tinued in a state of equilibrium, and product fractions
with a constant composition were recovered and analyzed.

Table 3 shows the volumes of the feeds, recycled

13

solution and product fractions, and Table 4 shows the compositions of molasses and the product fractions. Table 5 shows the colours of the molasses, residues and product fractions.

5   Table 3

        Volumes of feeds, recycled liquid and product fractions (1)

Step No.	1	2	3	4	5
Molasses feed	18	-	-	-	-
Water feed	33.3	5.0	13.0	34.0+10.0	-
Residue fraction from column 1	18	5.0	-	-	-
Residue fraction from column 2 or 3	-	-	-	34.0+10.0	-
Betaine fraction	-	-	13.0	-	-
Recycle fraction	7.3	-	-	-	-
Sucrose fraction	26.0	-	-	-	-
Recycled solution	-	6.0	26.0	-	44.0+5.0

20   Table 4

Compositions of feed and product fractions

	Dry solids (kg/l)	Sucrose (% by weight on d.s.)	Betaine (% by weight on d.s.)	Other substances (% by weight on d.s.)
Molasses feed	0.760	57.1	5.4	37.5
Residue fraction	0.069	18.7	6.8	74.5
Betaine fraction	0.048	5.3	47.5	47.2
Sucrose fraction	0.264	89.4	1.0	9.6

SUBSTITUTE SHEET

14

Table 5
Colours of molasses and product fractions
Example 1

	colour ICUMSA	residue 1	residue 2	residue 3
Molasses	47700			
Residue	115400	123600	151000	43324
Betaine	29900			
Sucrose	2100			

Example 2

	colour ICUMSA	residue 1	residue 2	residue 3
Molasses	38250			
Residue	92500	136000	240600	25900
Betaine	21800			
Sucrose	4300			

Example 3

A pilot plant scale chromatographic test apparatus was employed. The apparatus included three columns, feed pumps, recycling pumps, eluant water pumps, flow and pressure regulators, and inlet and outlet valves for the different process streams. The flowchart is shown in Figure 3.

The columns were packed with a strongly acid cation exchange resin ("Finex"). The resin had a poly-styrene/divinylbenzene backbone and was activated with sulphonic acid groups; the mean spherule size was about 0.36 mm. The resin had a DVB content of 5.5%. Initially the resin had been regenerated to sodium form, and during the run it was balanced with cations from the feed molas-ses.

15

Test conditions:

Diameter of column	200 mm
Height of resin bed:	
columns 1, 2 and 3	5000 mm
Temperature	75°C
Flow rates	22, 35, 40, 45, 70, 75 l/h

The feed solution consisted of beet molasses wherefrom calcium had been removed by adding sodium carbonate (pH 9) and filtering the precipitate off using diatomaceous earth as an aid.

The separation of sucrose and betaine was performed by a five-step sequence in which each column had its specific function. As shown in Figure 3, step 3 comprises one recycling phase and step 5 three recycling phases. The duration of the sequence was 111 minutes and the sucrose yield 81.9% (on the amount of sucrose fed).

Step 1: Molasses was fed into column 1 at a flow rate 35 l/h, and the recycling fraction and sucrose fraction were eluted from column 3.

Step 2: Water was fed into column 1 at a flow rate 70 l/h and the sucrose and recycling fractions were eluted from column 3.

Step 3: Recycling in column 1 at a flow rate 70 l/h; simultaneously water was supplied to column 2 at a flow rate 40 l/h and the betaine fraction was eluted from column 3.

Step 4: Water was fed into columns 1, 2 and 3 at flow rates 70, 75 and 40 l/h, the residue fractions were eluted from columns 1, 2 and 3, and the elution of the betaine fraction was continues from column 3.

Step 5: Recycling in columns 1, 2 and 3 at flow rates 22, 75 and 45 l/h.

After the sequence was completed, the process control program was continued and it returned to step 1. By repeating this sequence five to seven times, an

16

equilibrium was reached in the system. The run was continued in a state of equilibrium, and product fractions with a constant composition were recovered and analyzed.

Table 6 shows the volumes of the feeds, recycled solutions and product fractions, and Table 7 shows the compositions of the molasses and product fractions.

Table 6

Volumes of feeds, recycled liquid and product fractions (1)

Step No.	1	2	3	4	5
Molasses feed	20				
Water feed		20	10	20+26+20	
Residue fraction					
from column 1				26	
from column 2				26	
from column 3				15	
Betaine fraction			10	5	
Recycle fraction	9	4			
Sucrose fraction	11	16			
Recycled solution			8		11+12+11

Table 7

Compositions of feed and product fractions

	Dry solids (kg/l)	Sucrose (% by weight on d.s.)	Betaine (% by weight on d.s.)	Other substances (% by weight on d.s.)
Molasses feed	0.754	59.6	5.6	34.8
Residue fraction	0.081	16.7	8.8	74.5
Betaine fraction	0.071	45.9	22.9	31.2
Sucrose fraction	0.252	92.7	0.7	6.6

SUBSTITUTE SHEET

17

Claims:

1. A method for the fractionation of molasses using
a chromatographic simulated moving bed system in which
5   the liquid flow is effected in a single direction in a
system comprising at least two chromatographic partial
packing material beds, c h a r a c t e r i z e d   in
that the product or products are recovered during a
multi-step sequence comprising the following phases:
10   feeding phase of molasses, eluting phase and recycling
phase, wherein the liquid present in the partial packing
material beds with their dry solids profile is recycled
in the recycling phase in a loop comprising one, two or
several partial packing material beds.

15   2. A method as claimed in claim 1,   c h a r a c -
t e r i z e d   in that each loop comprises one whole
dry solids profile.

3. A method as claimed in claims 1 and 2,
c h a r a c t e r i z e d   in that a step comprises a
20   molasses feeding phase and/or one or more feeding phases
of an eluant liquid, and a recovery phase of a product
or products.

4. A method as claimed in claims 1 and 2,
c h a r a c t e r i z e d   in that a step comprises one
25   or more recycling phases, and a feeding phase for eluant
liquid and a product recovery phase.

5. A method as claimed in claims 1 to 4,
c h a r a c t e r i z e d   in that the product or prod-
ucts comprise a residue and sucrose and/or betaine.

30   6. A method as claimed in claims 1 and 2,
c h a r a c t e r i z e d   in that a step comprises two
or more recycling phases.

7. A method as claimed in claims 1 and 2,
c h a r a c t e r i z e d   in that a sequence comprises
35   five to ten steps.

18

8. A method as claimed in any one of claims 1 to 7, c h a r a c t e r i z e d in that a sequence comprising said steps is repeated five to seven times in order to reach an equilibrium in the system, and the method is continued with in the state of equilibrium reached.

9. A method as claimed in any one of claims 1 to 8, c h a r a c t e r i z e d in that a system comprising three to twelve, preferably three to six chromatographic partial packing material beds is employed.

10. A method as claimed in any one of claims 1 to 9, c h a r a c t e r i z e d in that a partial packing material bed is one column.

11. A method as claimed in any one of claims 1 to 10, c h a r a c t e r i z e d in that a strongly acid cation exchange resin is employed as a packing material for said columns.

12. A method as claimed in claim 11, c h a r a c - t e r i z e d in that said strongly acid cation exchange resin is in monovalent form, preferably in sodium or potassium form, or as a mixture of these forms.

13. A method as claimed in any one of claims 1 to 12, c h a r a c t e r i z e d in that the flow rate of the liquid in the columns is 0.5-10 $m^3/h/m^2$, even 20 $m^3/h/m^2$.

14. A method as claimed in any one of claims 1 to 13, c h a r a c t e r i z e d in that the temperature of the feed solution and eluant water is about 40-85°C, even 95°C.
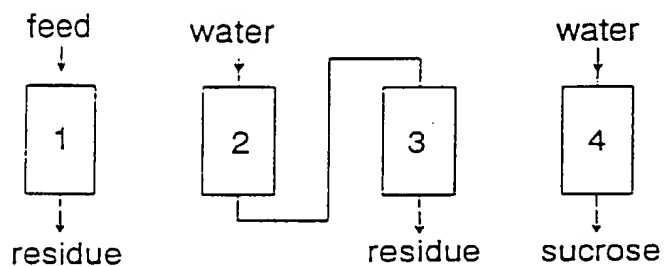
15. A method as claimed in any one of claims 1 to 14, c h a r a c t e r i z e d in that the dry solids content of the feed solution is 20-65% by weight, even 80% by weight.
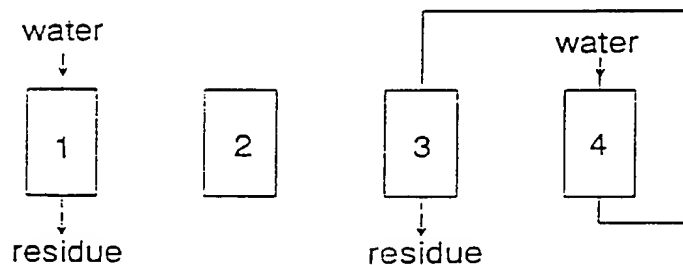
FIG. 1
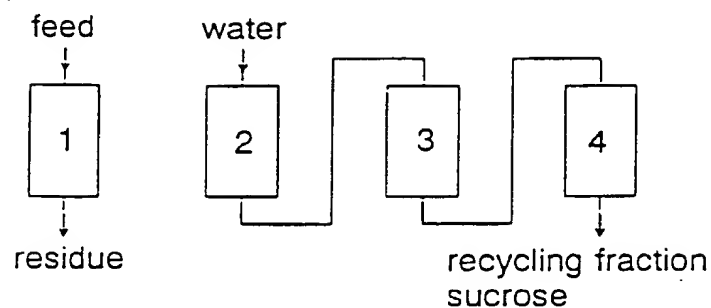
STEP 1

feed	18 l	(50 l/h)
residue	18 l	(50 l/h)
recycling		
fraction	7.5 l	(90 l/h)
sucrose	13.5 l	(90 l/h)

STEP 2

residue (1)	(cont'd)
(50 l/h)	
residue (3)	5.0 l
(90 l/h)	
sucrose	8.5 l
(90 l/h)	

STEP 3

| residue (1) | 4.0 l | (50 l/h) |
| residue (3) | 8.0 l | (90 l/h) |
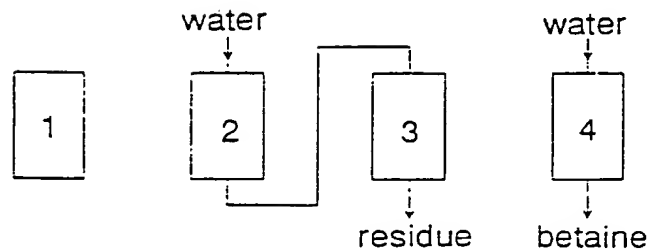
STEP 4

| residue | 5.0 l | (90 l/h) |
| betaine | 0 l | |

cont'd

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FIG. 1 (cont'd)

Loop I   Loop II

STEP 5
recycl. (loop I)   26 l
(90 l/h)
betaine 26 l
(70 l/h)

STEP 6
residue 25 l
(90 l/h)
recycl. (loop II) 20 l
(70 l/h)

STEP 7
recycl. (loop I) 26 l
(90 l/h)
residue 0 l

STEP 8
recycl. (loop I) cont'd
(90 l/h)
recycl. (loop II) 6 l
(40 l/h)

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FIG. 2

feed   water

| 1 | | 2 | | 3 |

residue   recycling fraction
sucrose
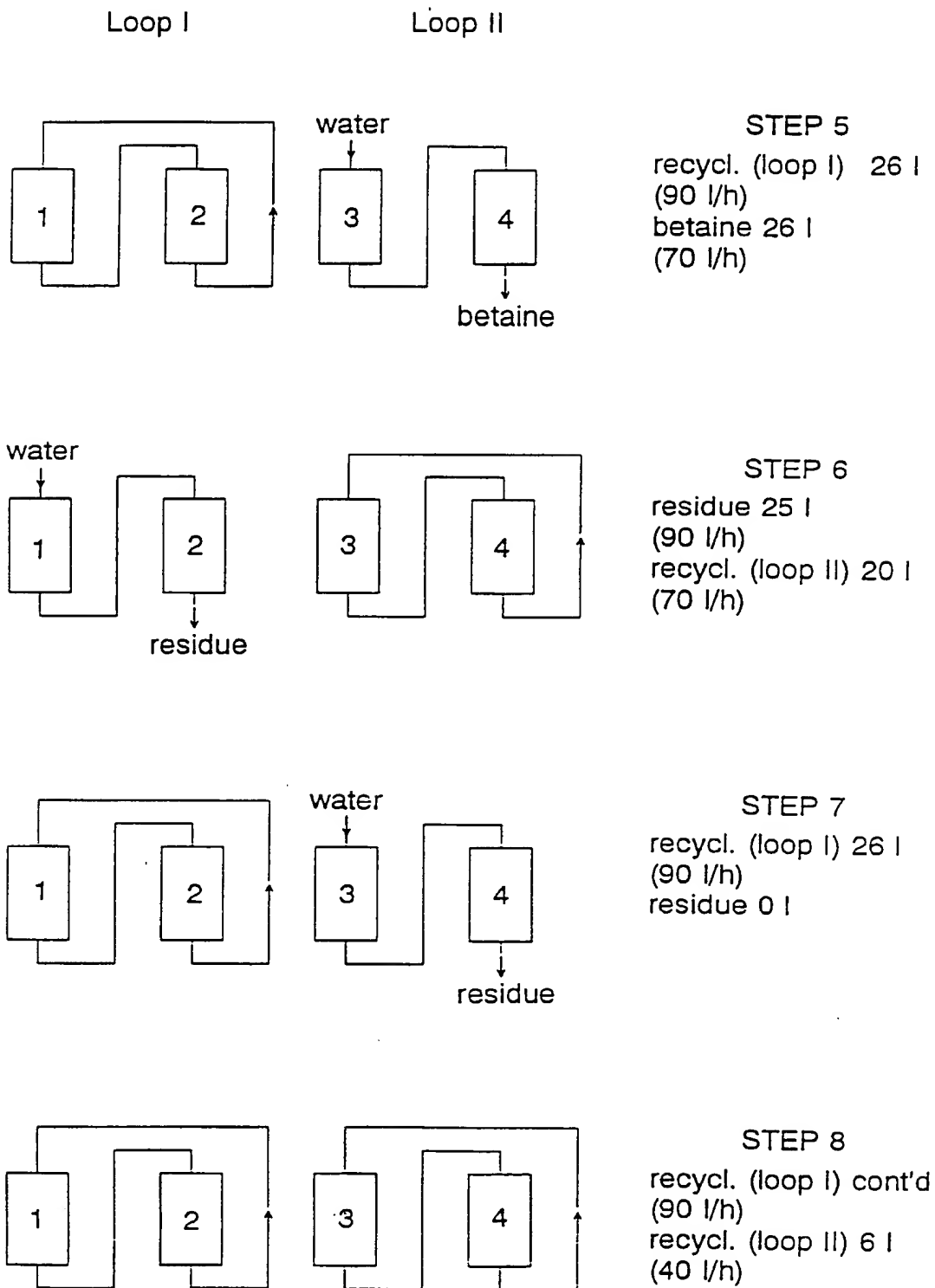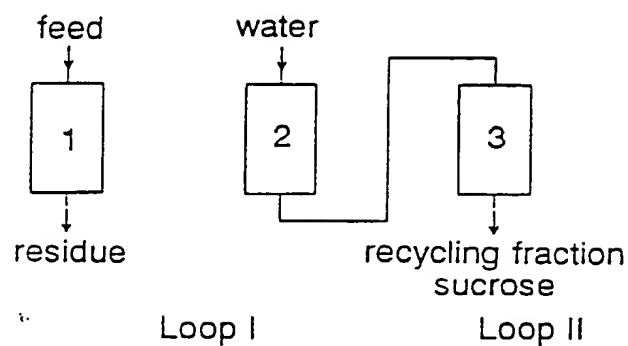
STEP 1

| feed | 18 l | (45 l/h) |
| residue | 18 l | (45 l/h) |
| recycling |
| fraction | 7.3 l | (85 l/h) |
| sucrose | 26 l | (85 l/h) |

Loop I   Loop II

water

| 1 | | 2 | | 3 |
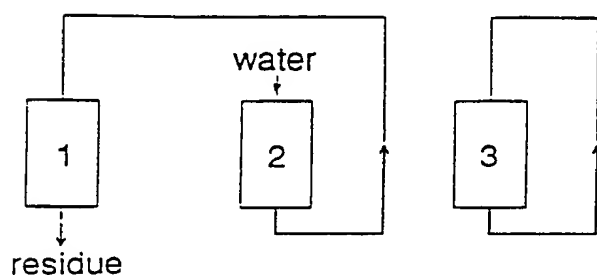
residue

STEP 2

residue (loop I) 5 l
(110 l/h)
recycl. (loop II) 6 l
(25 l/h)

water

| 1 | | 2 | | 3 |
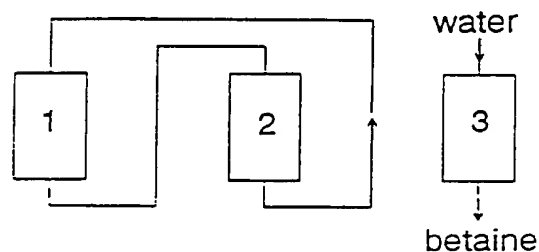
betaine

STEP 3

recycl. (loop I) 26 l
(110 l/h)
betaine 13 l
(35 l/h)

water   water

| 1 | | 2 | | 3 |
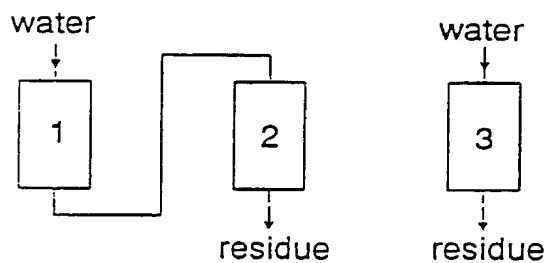
residue   residue

STEP 4

residue (loop I) 34 l
(110 l/h)
residue (loop II) 10 l

| 1 | | 2 | | 3 |
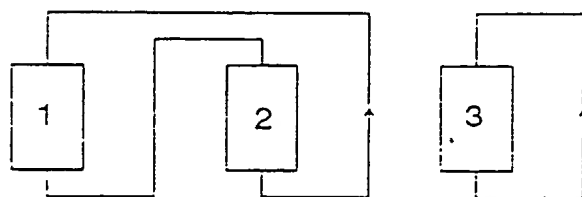
STEP 5

recycl. (loop I) 44 l
(110 l/h)
recycl. (loop II) 5 l
(25 l/h)

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FIG. 3
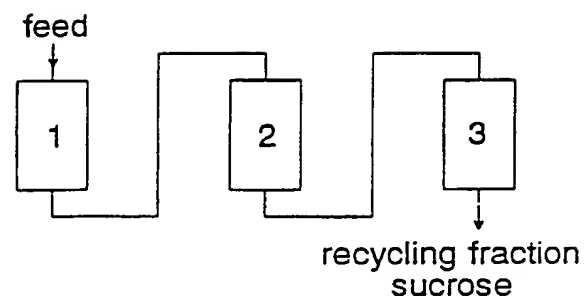
STEP 1

feed	20 l	(35 l/h)
recycling fraction	9 l	(35 l/h)
sucrose	11 l	(35 l/h)
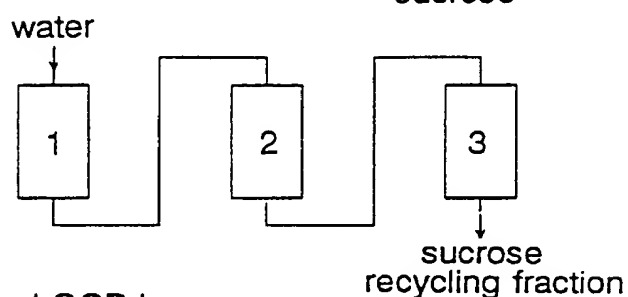
STEP 2

sucrose	16 l	(70 l/h)
recycling fraction	4 l	(70 l/h)
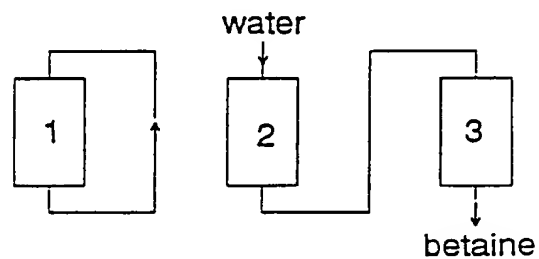
STEP 3

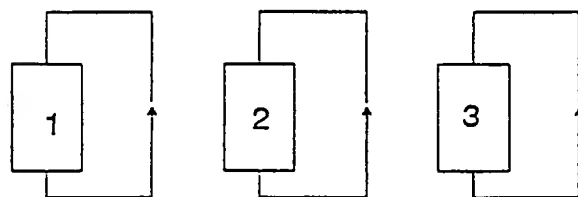| recycl. (loop I)	8 l	
(70 l/h)		
betaine		10 l
(40 l/h)		
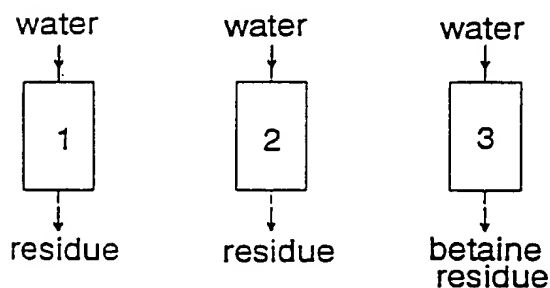
STEP 4

1. residue 26 l (70 l/h)
2. residue 26 l (75 l/h)
   betaine 5 l (40 l/h)
3. residue 15 l (40 l/h)

STEP 5

recycl. (loop I)	11 l
(22 l/h)	
recycl. (loop II)	12 l
(75 l/h)	
recycl. (loop III)	11 l
(70 l/h)	

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 94/00024

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C13D 3/14, B01D 15/08

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C13D, B01D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 5127957 (H. HEIKKILA ET AL), 7 July 1992 (07.07.92)	1-15
	--	
Y	US, A, 5102553 (M. M. KEARNEY ET AL), 7 April 1992 (07.04.92)	1-15
	--	
A	GB, A, 2240053 (JAPAN ORGANO CO LTD), 24 July 1991 (24.07.91)	1-15
	--	

☐ Further documents are listed in the continuation of Box C.   ☒ See patent family annex.

*	Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	
"E"	earlier document but published on or after the international filing date	"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	
"O"	document referring to an oral disclosure, use, exhibition or other means	"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"P"	document published prior to the international filing date but later than the priority date claimed	"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 April 1994	16 -05- 1994
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Carolina Gomez Lagerlöf Telephone No. +46 8 782 25 00

Form PCT/ISA/210 (second sheet) (July 1992)

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US-A- 5127957	07/07/92	AU-A-	3500289	14/12/89
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		EP-A-	0345511	13/12/89
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		JP-T-	4502276	23/04/92
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		AU-A-	6908291	18/07/91
		WO-A-	9108815	27/06/91
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		FR-A,B-	2656231	28/06/91
		JP-A-	4227804	17/08/92
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